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PREPARATION AND PROPERTIES OF THIN FILM REVERSIBLE
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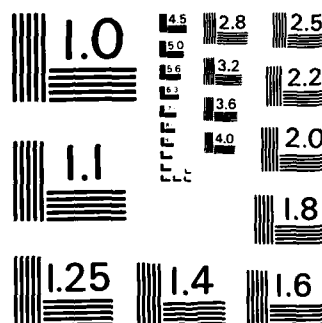
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**PREPARATION AND PROPERTIES OF THIN FILM
REVERSIBLE CATHODE MATERIALS**

FINAL REPORT

**Bruce Dunn and R. F. Bunshah
Department of Materials Science and Engineering**

July 1985

Contract/Grant Number: DAAG29-82-K-0145

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School of Engineering and Applied Science
Los Angeles, CA 90024**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The research program clearly established that a reversible cathode material with a highly anisotropic structure could be prepared as a thin film, 2 to 5 μ m thick. The research emphasized the synthesis of TiS_2 by the evaporation of titanium in a plasma-enhanced H_2S gas. Nearly-stoichiometric films of TiS_2 were produced. The electrical conductivity and certain electrochemical properties of the films were comparable to the bulk material. Lithium diffusion coefficients were found to be lower than the bulk material and dependent upon film composition. Character supplied keywords include:		

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1. FOREWORD

This report pertains to the research undertaken and accomplished over the period June 1, 1982 - May 31, 1985. The technical materials mentioned herein were more fully described in the five (5) previous semi-annual progress reports submitted to ARO. The progress achieved over the final project period of 5 months (January 1, 1985 - May 31, 1985) is included in this report. The experimental work during this time primarily involved electrochemical measurements, and the results are contained in the appropriate section of this report.

At the time of this writing, there were a few items in progress associated with project. They are as follows;

1. A small number of electrochemical measurements are continuing. These experiments are either confirming in nature or of a more prolonged duration (i.e., cell cycling). This work is being used to satisfy and complete a student's M.S. thesis.
2. The M.S. thesis of Mr. D. A. Zehnder is in preparation. The date of completion is scheduled for September 1985.
3. A manuscript based on the thesis of Mr. Zehnder (but not necessarily on the experiments in progress) is also in preparation. The paper will be submitted as part of the conference proceedings for the International Conference on Solid State Ionics in August 1985.

Items (2) and (3) above are listed in the text of this report for the sake of completeness. The fact that they are in preparation is clearly identified. Upon completion, these items will be sent to ARO in accordance with the usual procedures.

Finally, the authors of this report are very grateful to the U.S. Army Research Office for the support of this research project and for their continued support of our work on thin film cathode materials.

2A. STATEMENT OF THE PROBLEM STUDIED

The overall objective of the research was to synthesize reversible cathode materials in thin film form. Such materials had not been previously prepared as thin films and it was unknown whether (a) vapor deposition approaches could be used to synthesize compounds with highly anisotropic structures and (b) whether such materials would display fundamentally different properties as a thin film compared to the bulk material. Thin films also offer some interesting possibilities for secondary lithium batteries including a shorter path length for ion and electron transport and novel cathode structures. The reversible cathode material titanium disulfide (TiS_2) was emphasized because considerable information concerning the preparation, structure and properties of the bulk material was available to provide valuable comparisons with the thin films. The breadth of the research program included the preparation of thin films by vapor deposition processes, the characterization of the structural and chemical nature of the films and the investigation of their transport properties and electrochemical behavior.

2B. SUMMARY OF THE MOST IMPORTANT RESULTS

a. Film Synthesis and Characterization

1. A reversible cathode material with a highly anisotropic structure has been prepared as a thin film (2 to 5 μm thick) using vapor deposition processes.
2. The ARE process (activated reactive evaporation) offers considerable flexibility in controlling the chemistry of the film, the resulting crystal phase and the microstructure of the film.
3. Under optimized conditions, nearly stoichiometric films ($\text{Ti}_{1.04}\text{S}_2$) can be produced. The films may also show preferential orientation, with the basal planes of TiS_2 parallel to the plane of the substrate.
4. The films are of high quality. They are free of pin holes and defects and adhere extremely well to a variety of glass and ceramic substrates.

b. Transport Properties and Electrochemical Behavior

This category is further divided depending upon whether the property of the film is similar or not to the corresponding bulk material.

Properties similar to the bulk material

1. For as-deposited films of TiS_2 , the electrical resistivity values at room temperature and the temperature dependence of the resistivity (increasing resistivity with increasing temperature) are in good agreement with the behavior reported for bulk TiS_2 .
2. The films are electrochemically active. Prior to the actual testing, however it is best to "condition" the film by lithiating it. The lithiation is accomplished by a chemical approach and vastly improves the electrochemical performance. Similar procedures are frequently employed for bulk materials.
3. The films are able to electrochemically intercalate and de-intercalate lithium. The operating voltage range (2.7V to 1.7V) is comparable to that of the bulk material and cycling occurs at the $\mu\text{A}/\text{cm}^2$ level. No long term cycling studies were performed.
4. The electrochemical measurements gave no evidence of interfacial anomalies or phase transformations.

Properties different than the bulk material

5. The diffusion coefficient for lithium was some 10^2 to 10^3 times less than the bulk material.
6. The diffusion coefficient for lithium varied as a function of the lithium content of the TiS_2 film. The diffusion coefficients were highest over the voltage range 1.8V - 2.3V and substantially lower for those films with $x < 0.1$ (for the composition Li_xTiS_2). In contrast, bulk TiS_2 exhibits no such variation in the lithium diffusion coefficient over the complete range $0 < x < 1$.

7. The different transport properties for the TiS_2 thin films are not altogether unexpected. The materials are titanium rich in their stoichiometry, may exhibit preferred orientation of crystallites and often contain a sizeable fraction of an amorphous phase. Each of these features would serve to impede lithium diffusion as compared to bulk materials.

2C. LIST OF PUBLICATIONS AND TECHNICAL ITEMS

1. Publications

'Preparation and Characterization of TiS_x Films Using Activated Reactive Evaporation,' by E. Zeira, C. Deshpandey, R. Budhani, H. Doerr, D. Zehnder, R. Bunshah and B. Dunn, submitted to Journal of Vacuum Science and Technology (manuscript attached).

'Transport Properties of Thin Film TiS_2 ' by D. Zehnder, C. Deshpandey, B. Dunn and R. Bunshah, in preparation, to be submitted to Solid State Ionics.

2. Presentations

'Preparation and Characterization of TiS_x Films Using the ARE Technique' by E. Zeira et al. Presented at the International Conference on Metallurgical Coatings, San Diego, CA, April 1984.

'Preparation of TiS_2 Films Using the ARE Technique,' by E. Zeira et al. Presented at the American Vacuum Society Meeting, Reno, NV, November 1984.

'Transport Properties of Thin Film TiS_2 ,' by D. Zehnder et al. To be presented at the International Conference on Solid State Ionics, Lake Tahoe, NV, August 1985.

3. Theses

'Preparation and Properties of Thin Film Reversible Cathode Materials.'
M.S. Thesis by E. Zeira, December, 1984.

'Transport and Electrochemical Properties of Thin Film TiS_2 .' M.S.
Thesis by D. Zehnder, in preparation, to be submitted September, 1985.

2D. PARTICIPATING SCIENTIFIC PERSONNEL AND ADVANCED DEGREES AWARDED

Dr. R. F. Bunshah

Dr. C. V. Deshpandey

Dr. B. Dunn

Mr. D. A. Zehnder (M.S. degree to be awarded, September, 1985)

Mr. E. C. Zeira (M.S. degree awarded December, 1984)

APPENDICES

APPENDIX I: A copy of the manuscript, 'Preparation and Characterization of TiS_x Films Using Activated Evaporation Techniques

APPENDIX II: The title page and abstract of the M.S. Thesis of E. C. Zeira

Preparation and Characterization of TiS_x
Films Prepared Using
Activated Reactive Evaporation Techniques

E. C. Zeira, C. V. Deshpandey, R. C. Budhani,
H. J. Doerr, R. F. Bunshah, D. Zehnder and B. Dunn

School of Engineering and Applied Science
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stract

TiS_2 has been the subject of intense study because of its superior performance as a reversible cathode in lithium electrochemical cells. Thin films of nearly stoichiometric TiS_2 were deposited by evaporating Ti metal in an H_2S atmosphere. The effect of deposition conditions such as deposition rate, H_2S partial pressure and flow rate, and substrate temperature on the composition of the TiS_2 films was analyzed using EDAX, while x-ray diffraction and electron diffraction techniques were used to identify the crystalline phases present. The conditions required to synthesize fine-grained crystalline TiS_2 films are discussed.

Introduction

The phenomenon of fast ion transport in solids has led to the study of several new types of rechargeable battery systems which are capable of possessing high energy densities^(1,2). The use of lithium as an anode material particularly attractive and numerous ionic conductors have been considered reversible cathode materials for lithium batteries. It is generally believed that a successful cathode should incorporate the anode element into its structure. Intercalation reactions are ideal for this purpose since the process involves the insertion of a guest species into the host lattice without causing structural alteration. The reversible nature of this reaction has enabled transition metal dichalcogenides (among other materials) to be used successfully as cathodes in electrochemical cells. In addition to reversibility, solid state cathode materials should also exhibit a number of other properties including good electronic and ionic conductivities, a large free energy of reaction with lithium, a wide composition range and appropriate structural stability with intercalated lithium, and chemical stability in organic electrolytes⁽²⁾.

Transition metal dichalcogenides have received considerable attention for reversible cathode applications. These materials characteristically exhibit layered structures in which alternate layers of anions are held together by weak van der Waals forces. The van der Waals gap between layers offers sufficient vacant sites to permit a wide composition range of intercalated lithium as well as convenient pathways for rapid lithium diffusion. Titanium

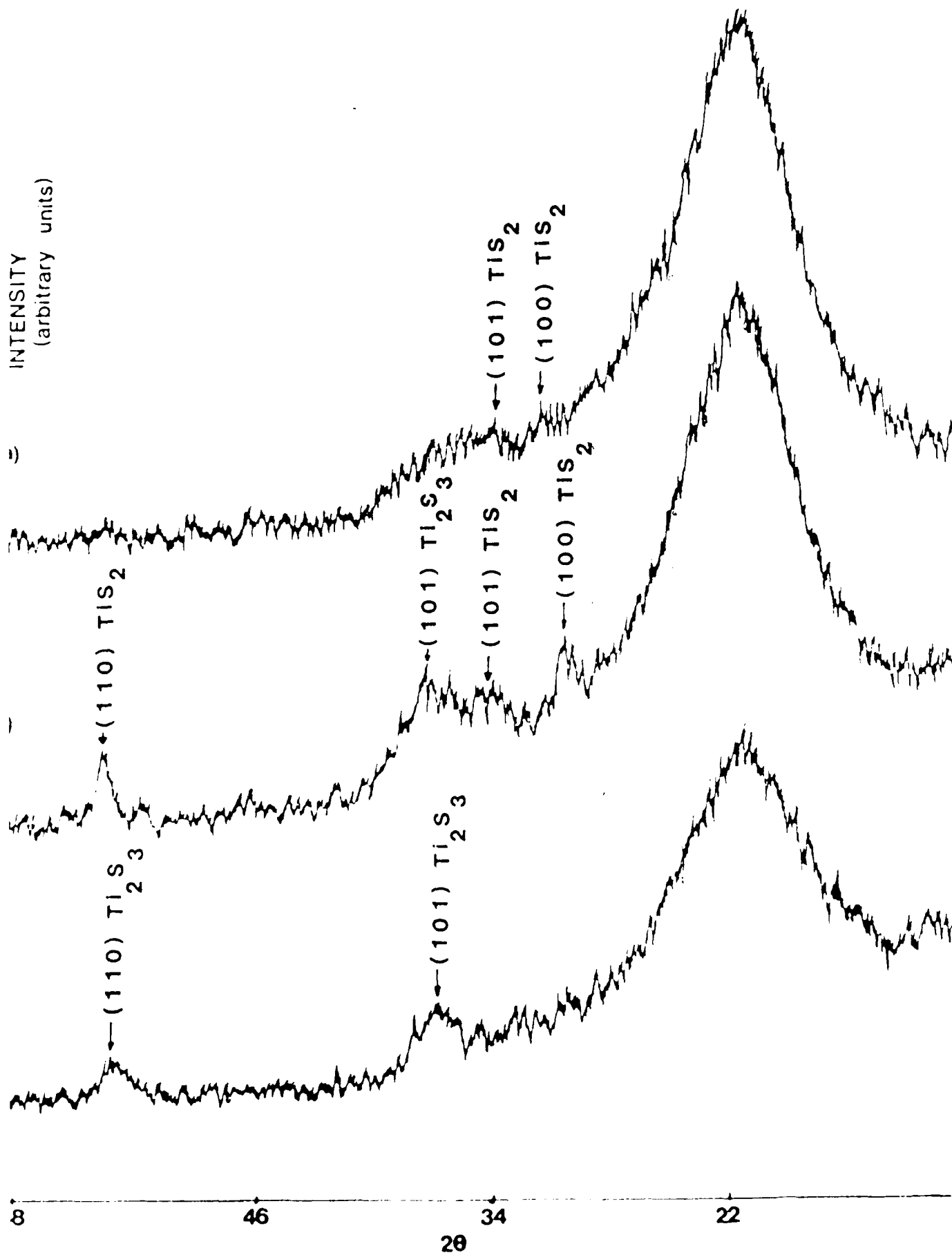


Figure III

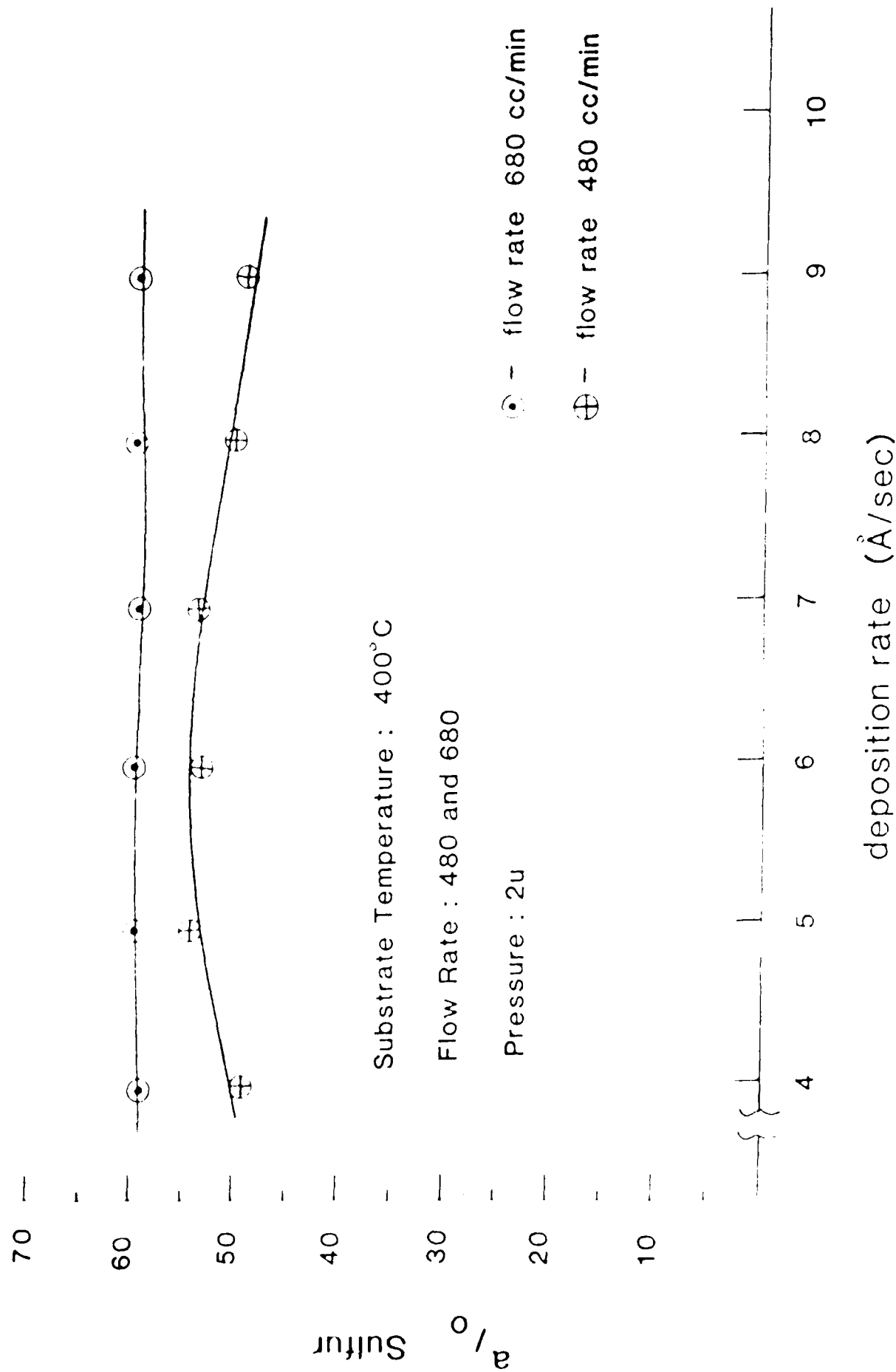


Figure 11

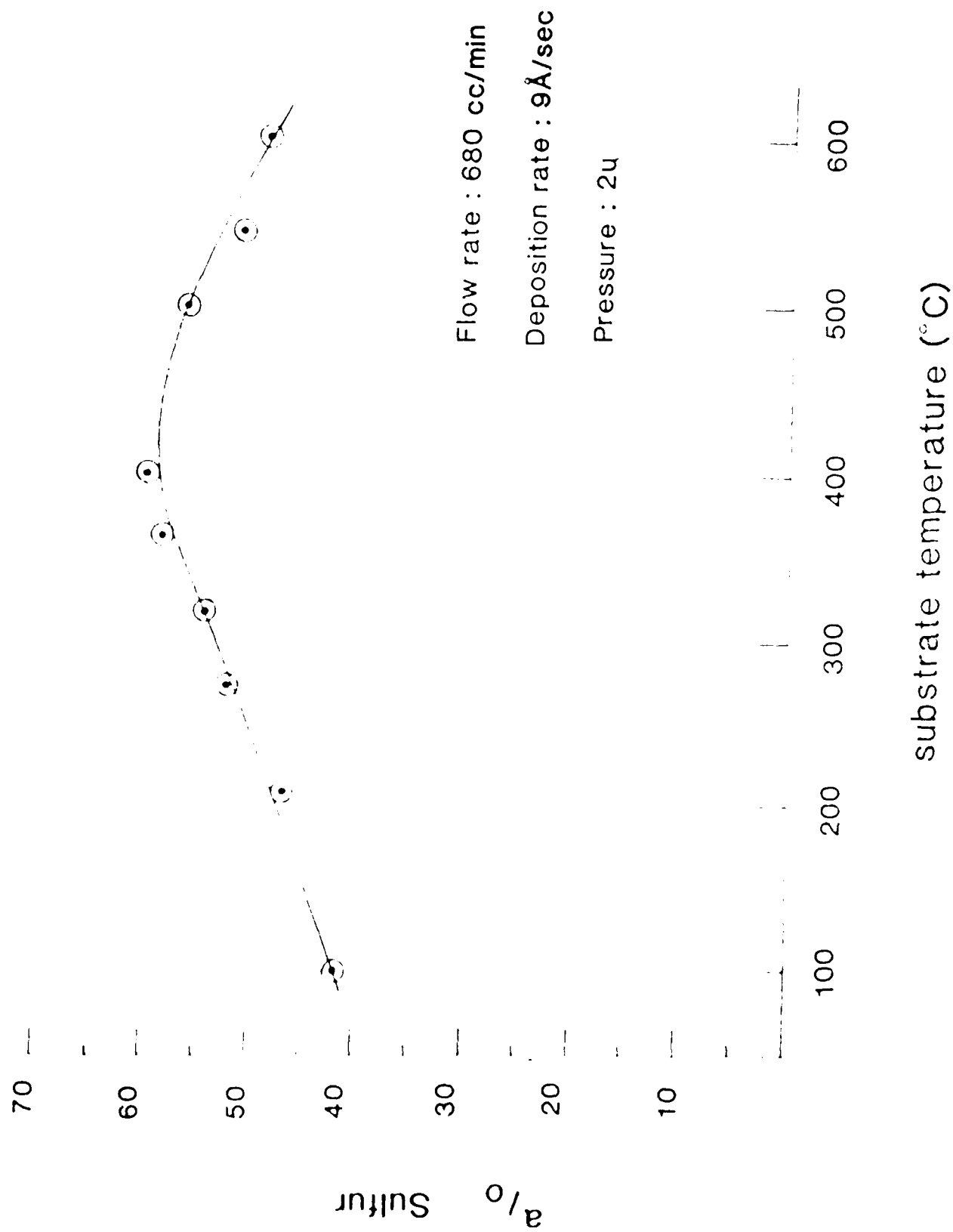


Figure 1

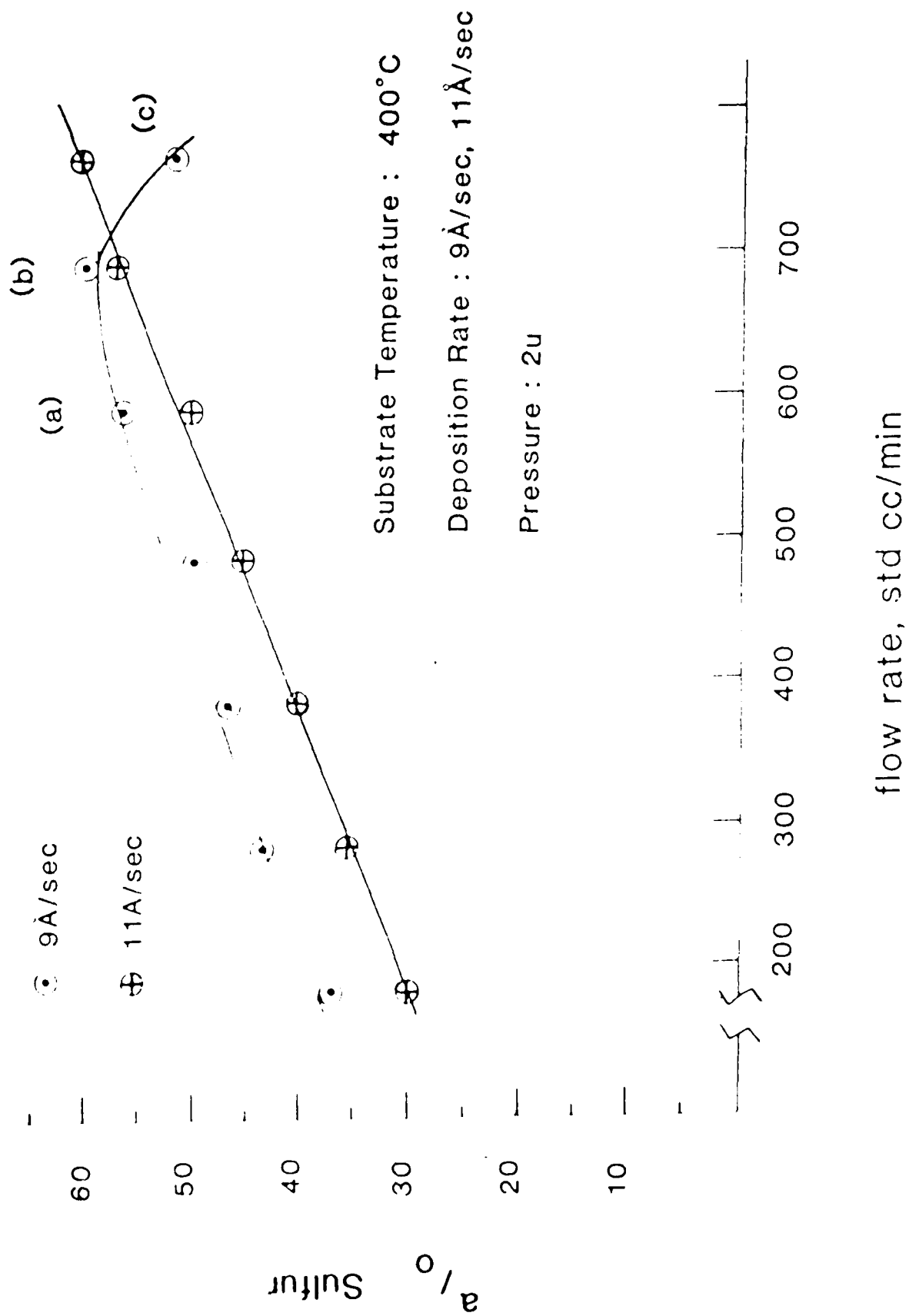


Figure Captions

Figure I: Atomic Percent Sulfur vs. Flow Rate

Figure II: Atomic Percent Sulfur vs. Deposition Rate

Figure III: Atomic Percent Sulfur vs. Substrate Temperature

Figure IV: X-Ray Diffraction Patterns for TiS_x Samples From Figure 1

TABLE I

Phases present in films based on electron diffraction analysis.

<u>Dep. Rate</u>	<u>H₂S flow</u>	<u>Subst. temp</u>	<u>Phase</u>
^o 540A/min	580 SCCM	400°C	TiS ₂
^o 540A/min	680 SCCM	400°C	Ti ₂ S ₃ + TiS ₂
^o 540A/min	780 SCCM	400°C	Ti ₂ S ₃
^o 540A/min	600 SCCM	350°C	TiS ₂ + Ti ₃ S ₄
^o 540A/min	600 SCCM	500°C	Ti ₂ S ₃
^o 540A/min	600 SCCM	600°C	Ti ₃ S ₄

VI. References

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IV. Conclusions

Various titanium sulfide phases, including a nearly stoichiometric TiS_2 phase, were deposited using the ARE technique. The phases, film stoichiometry and structure were found to be quite sensitive to deposition conditions.

The flow rate of the H_2S gas, combined with the evaporation rate of titanium, were found to have the most profound effects on the percent sulfur and phases found in the films. These parameters were found to be inversely related to one another and hence, a maximum in the percent sulfur was realized. Different phases were produced corresponding to the amount of sulfur (determined by the flow rate of the gas and the $\text{H}_2\text{S}/\text{H}_2$ ratio) and the amount of Ti available for the reaction. A high evaporation rate and a low H_2S flow rate gave the low sulfide phase while a lower evaporation rate at a higher flow rate gave the higher sulfide phase. The temperature of the substrate influenced the films to a lesser degree. An optimum condition was found to cover a wide range of temperatures (300-440°C). Hence, less precise control over this parameter was needed. This situation occurred since the substrate temperature was not the sole source of the kinetic energy. The plasma also contributed to the kinetic energy of the constituents, making the species more energetic and thereby activating the reaction.

V. Acknowledgements

This research was supported by the U.S. Army Research Office.

produces the observed maxima in %S displayed in Fig. 1 and Fig. 2. Our experiments show that ratio of H_2S/H_2 in the plasma controls the stoichiometry of the films. Detailed analysis of this behavior based on emission spectroscopic studies of the plasma will be presented in a subsequent publication.

B. Phases present in the deposit

The effects of deposition conditions on the phases present in the films are indicated in Table I. Fig. 4 shows X-ray diffraction patterns for films deposited under three different conditions as indicated by the letters a, b, c displayed in Fig. 1. The large peak at $2\theta = 22^\circ$ is diffraction from the Vycor substrate. In sample (a), the TiS_2 phase has a very small grain size and electron diffraction suggests a (100) preferred orientation. Structure of the film is not obvious from the X-ray results (Fig 4-a). Sample b (Fig. 4-b) shows a larger grain size (note the intensity of the peaks) and still exhibits a (100) preferred orientation. In addition two more peaks appear in the diffraction patterns corresponding to the (101) reflection of Ti_2S_3 and the (110) reflection of TiS_2 . This suggests that sample (b) contains two phases, Ti_2S_3 and TiS_2 . Abendroth et al.⁽⁸⁾ have also reported the existence of a two phase region of TiS_2 and Ti_2S_3 below $500^\circ C$. Sample (c) (Fig. 4-c) shows the pattern of Ti_2S_3 single phase film. Based on these results, one may conclude that for a given deposition rate and substrate temperature, the phases present in the films depend on H_2S flow rate. A similar dependence of the phases present in the films is observed with substrate temperature as indicated in Table I.

500°C, the equilibrium sulfur pressure above TiS_2 rises dramatically with temperature. For example, at 500°C, the sulfur pressure above TiS_2 is 76 torr and climbs to about 7 atm. at 600°C. Since the working pressure in the chamber does not exceed 1×10^{-2} torr, one would expect to see a decrease in the sulfur content of the films at temperatures above 500°C. At temperatures below 300°C, the rate of reaction is slower due to desorption of sulfur from the substrate. Desorption occurs because of the high vapor pressure of sulfur, H_2S and HS at relatively low temperature. Thus, the mean residence time of these species (S, H_2S , HS) on the substrate is very short at such temperatures. Consequently, these species desorb from the substrate prior to reacting with Ti, leaving sulfur deficient films. This combination of features explains why there are sulfur deficient films at high and low temperature, with a maximum at 400-425°C.

Apart from the rate of arrival of Ti and S on the substrate, the experimental conditions which determine the species present in the plasma contribute significantly to the observed variation in the %S as shown in Figs. 1 and 2. During the deposition process, the plasma dissociates the H_2S molecule and leads to the formation of species such as H_2S^+ , HS^- , H_2 and S_2 . The type of species produced depends on electron-molecular collisions (defined in terms of collision cross-section) that take place in the region between the source and the substrate. For a given energy (anode potential, 100-110V in our case) the collision cross-section depends on the number of H_2S molecules present and the electron density (number of electrons/unit vol). The number of H_2S molecules is related to H_2S flow rate whereas electron density is related to evaporation rate which controls the number of secondary electrons in the plasma. It is this relationship between the H_2S flow rate and evaporation rate which

expected that the deposition rate curve would exhibit a distinct maximum in %S, analogous to that observed in Fig. 1 for H_2S flow rate. However, we believe that the flow rates used were too high to produce a maximum for the deposition rates employed.

The sulfur content of the films showed an increase with substrate temperature up to about 400°C and then decreased somewhat when temperature was increased further. A typical variation in %S with substrate temperature for a constant deposition rate and H_2S flow rate is shown in Fig. 3.

The observed variation in %S shown in Figs. 1, 2 and 3 indicates that there exists a definite relationship among the deposition parameters (i.e., deposition rate, flow rate and substrate temperature) which controls the stoichiometry of the films. To obtain a high sulfur content in the films, there must be a specified number of Ti and S atoms arriving at the substrate and a sufficient amount of energy to activate the reaction. The number of titanium and sulfur atoms arriving at the substrate (per unit area per unit time) for a given pressure are directly proportional to the evaporation rate and H_2S partial pressure, respectively. At the same time the activation energy is dependent on the plasma conditions (which influences the kinetic energy of the depositing species) and the substrate temperature. Since plasma conditions were kept relatively constant during each experiment, the activation energy was primarily dependent on the substrate temperature. This is illustrated in Fig. 3 which shows that the sulfur content in the film varies with the substrate temperature. At the high temperature end of the graph, the percent sulfur decreases slightly. This phenomenon has also been observed in a number of prior investigations involving the preparation of $TiS_2^{(1-3)}$. Above

soda-lime glass and quartz substrates developed cracks on cooling due to thermal expansion mismatch.

B. Film Characterization

The films were analyzed for sulfur content (in atomic percentage) using Energy Dispersive Analysis (EDAX). A TiS_2 standard was used for calibration. The Scanning Electron Microscope (SEM) was also used to study the film surface morphology. Phase identification and lattice parameters were determined using JEOL CX STEM in the diffraction mode. The electron diffraction results were confirmed by X-ray diffraction techniques using a Norelco X-ray diffractometer and a Guinier X-ray camera.

III Results and Discussion

The film composition was found to be influenced by the H_2S flow rate, substrate temperature and deposition rate. The variation in %S in the film with respect to these parameters (keeping the other conditions constant) is shown in Fig. 1-3 respectively.

It can be seen from Fig. 1 that the H_2S flow rate plays a dominant role in establishing the sulphur content in the film. The amount of sulphur in the film varies as much as 30% between the minimum (200 cc/min) and maximum (800 cc/min) values of the flow rate. The dashed line shows that a higher deposition rate causes the maximum to be shifted towards a greater flow rate.

As shown in Fig. 2 the deposition rate did not produce a very pronounced effect on %S in the films for the range of flow rates investigated. It was

process, high energy species such as ions and high energy neutrals generated by neutral atom-ion collisions enhance the reactivity of the condensing species, i.e. the "activity" of the process. Hence compounds can be formed at relatively low pressures of the reactive gas⁽⁷⁾ as well as at low deposition temperatures. Considering this advantage, we have investigated the synthesis of TiS_2 by the ARE technique where Ti was evaporated in a H_2S plasma. The effect of deposition conditions such as deposition rate, H_2S flow rate and pressure, and substrate temperature on composition and structure of the film was investigated. The results of this investigation are reported below.

II. Experimental Procedure and Measurement Techniques

The films were deposited by evaporating Ti using an electron beam source in a H_2S plasma. The plasma was generated by accelerating the electrons thermionically emitted from a thoriated tungsten filament towards an anode plate maintained at 110 volts positive d.c. potential. The plasma was confined to the area between the substrate and the source using ceramic bar magnets arranged in a cylindrical configuration around the source. The H_2S flow rate was controlled by a flow meter while the required pressure ($\approx 2 \times 10^{-3}$ torr) was maintained by throttling the high vacuum valve. The deposition rate was measured using a quartz crystal located near the substrate and connected to a AIRCO Temescal (Model FDC-8000) thickness monitor. Substrates were located 12" above the source and were heated by quartz infrared lamps. The substrate temperature was monitored by a chromel-alumel thermocouple attached to the back of the substrate. A variety of substrates were used including soda-lime glass, quartz, polycrystalline alumina, and sapphire wafers. Alumina and sapphire substrates were found to give the best results. Films deposited onto

disulfide (TiS_2) is probably the most widely investigated transition metal dichalcogenide as it successfully fulfills most of the requirements for reversible cathode operation in lithium secondary batteries.

The conventional method of synthesizing TiS_2 is by direct reaction of the elements at elevated temperature (600°C)^(3,4). Standard pressed-powder cathodes are then prepared from this material. Although the performance of these cathodes is quite good, there are certain areas where improvements in the properties of TiS_2 would be highly desirable. The application of thin-film vacuum deposition methods to the fabrication of reversible cathode materials appears to offer several advantages. The use of thin films reduces the path length of charge transport, thus improving the kinetics of ion transport and lowering the electronic resistance. Thin film deposition techniques also offer novel microstructures, less contamination and they enable new battery configurations to be considered.

Synthesis of TiS_2 using vacuum deposition techniques was first reported by Perry⁽⁵⁾. Ti films about 3000 \AA thick were prepared by sputtering and subsequently heat-treated at 630°C in a H_2S ambient. The films produced were found to be $\text{Ti}_{1.005}\text{S}_2$. However this technique does not differ significantly from the direct reaction method mentioned earlier. More recently, Kanehori et al.⁽⁶⁾ prepared TiS_2 films by (VD) methods. In this case the microstructure consisted of narrow platelets which were fairly porous. Rather low diffusion coefficients for lithium were reported. In contrast to prior work, the Activated Reactive Evaporation (ARE) process⁽⁷⁾, which is extensively used to synthesize refractory compounds, transparent conductors, and superconducting compounds, appears to be an ideal technique for the synthesis of TiS_2 . In this

UNIVERSITY OF CALIFORNIA

Los Angeles

*Preparation and Properties
of Thin Film Reversible Cathode Materials*

A thesis submitted in partial satisfaction of the
requirement for the degree Master of Science
in Engineering

by

Eitan Chaim Zeira

1984

ABSTRACT OF THE THESIS

Preparation and Properties of Thin Film Reversible Cathode Materials

by:

Eitan Chaim Zeira

Master of Science in Engineering

University of California, Los Angeles, 1984

Professor R. F. Runshah, Committee Co-Chair

Professor Bruce S. Dunn, Committee Co-Chair

The binary phase of TiS_2 has been the subject of intense study because of its superior performance as a reversible cathode in lithium electrochemical cells. Thin film deposition offers several advantages over the conventional method in preparing this cathode material. In this investigation, thin films of TiS_2 were prepared using the Activated Reactive Evaporation (ARE) technique.

The films were deposited by evaporating Ti metal in an H_2S plasma. The effect of deposition conditions such as evaporation rate, H_2S flow rate and substrate temperature on the composition and structure of the films was studied. The films were characterized by X-ray diffraction, electron diffraction, energy dispersive X-ray analysis (EDAX), and scanning electron microscopy.

It was found that the ARE process can be used to deposit films of TiS_2 . The H_2S flow rate and the evaporation rate of titanium were found to be the more significant parameters that determine the composition and structure of the films.

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